

# Fabrication, Mechanical Characterization and Wear Response of Hybrid Composites Filled with Red Mud: an Alumina Plant Waste

Hemalata Jena<sup>1</sup>, Alok Ku. Satapathy<sup>2</sup>

<sup>1</sup>Department of Mechanical Engg., Indian Institute of Technology, Bhubaneswar, India-751013  
hemalatajena@iitbbs.ac.in

<sup>2</sup>Department of Mechanical Engg., National Institute of Technology, Rourkela, India-769 008  
alok.satapathy@gmail.com

**Abstract**—With increased environmental threat it has become necessary to find out alternative uses of industrial wastes and to develop value added products using them. This work is a step in that direction. A possibility that the incorporation of both particles and fibres in polymer could provide a synergism in terms of improved properties. In view of this, the present piece of research is undertaken which includes the fabrication of a set of glass-polyester composites using red mud an alumina plant waste product as the particulate filler. It also attempts to study the solid particle erosion wear response of these composites under multiple impact condition. The methodology based on Taguchi's experimental design approach is employed to make a parametric analysis of erosion wear process. This systematic experimentation has led to determination of significant process parameters and material variables that predominantly influence the wear rate of the particulate filled composites reinforced with glass fibre. This work, therefore proposes a correlation derived from the results of Taguchi experimental design. Using Taguchi method for analysis, the significant control factors predominantly influencing the wear rate are identified. The filler content in the composites, the impingement angle and impact velocity are found to have substantial influence in determining the rate of material loss from the composite surface due to erosion.

**Index Terms**—Hybrid Composite, Red Mud, Erosion Wear Rate, Taguchi Method

## I. INTRODUCTION

Production of alumina from bauxite by the Bayer's process is associated with the generation of red mud as the major waste material in alumina industries worldwide. Depending upon the quality of bauxite, the quantity of red mud generated varies from 55-65% of the bauxite processed [1]. Detailed characterization of red mud generated from NALCO aluminium refinery at Damanjodi, India is reported by Mohapatra et al. [2] and of some other sources by various authors [3]. To obtain the desired properties from a hybrid composite system, reinforcement and fillers are added to the polymers [4]. The additional improvements in mechanical and tribological properties are in many cases attained through the incorporation of glass or carbon fibre reinforcement and through the filling of particulate matters. Red mud is ceramic rich industrial waste and has potential to be used as filler

material in various polymer matrices. Hard particulate fillers consisting of ceramic or metal particles and fibre fillers made of glass are being used these days to dramatically improve the wear resistance, even up to three orders of magnitude [5]. The present research work has been undertaken, with an objective to modify its mechanical and erosion wear performance of the resulting composites with red mud of different filler loading condition.

## II. EXPERIMENTAL DETAILS

### A. MATERIALS

Unsaturated isophthalic polyester resin (density 1.35g/cc, elastic modulus 3.25 GPa) supplied by Ciba-Geigy of India is the matrix material in the present investigation. Red mud used as the filler material in this work has been collected from the site of Vedanta alumina plant Lanjigarh in India. It is alkaline, thixotropic and possesses a true density of 3.3 g/cc. Chemical analysis of red mud used in composite making for this investigation suggests its composition as : Fe<sub>2</sub>O<sub>3</sub> 44.6%, Al<sub>2</sub>O<sub>3</sub> 15.6% , SiO<sub>2</sub> 15.4% , TiO<sub>2</sub> 8.5% , Na<sub>2</sub>O 6.7% % and traces of CaO and MgO. In the present work, woven roving E-glass fibers (supplied by Saint Gobain Ltd. India) have been used as the reinforcing material. The major constituents of E-glass are silicon oxide (54 wt %), aluminum oxide (15 wt %), calcium oxide (17 wt %), magnesium oxide (4.5 wt %) and boron oxide (8 wt %). E-glass fiber has an elastic modulus of 72.5 GPa and possess a density of 2.59 gm/cc.

### B. COMPOSITE FABRICATION

The fabrication of the composite slabs will be done by conventional hand-lay-up technique with (D, E, F, G, H) and without (A, B, C) fibre mats followed by light compression moulding technique. The varying composition and the composite designations have to taken as shown in the Table 1. The fillers will mixed thoroughly in the polyester resin before the respective fibre mats are reinforced into the matrix body. The composite slabs are made by conventional hand-lay-up technique. 2% cobalt naphthalate (as accelerator) is mixed thoroughly in the polyester resin and then 2% methyl-ethyl-ketone-peroxide (MEKP) as hardener is mixed in the resin prior to reinforcement. Each ply of fibre is of dimension

200 × 200 mm<sup>2</sup>. A stainless steel mould having dimensions of 210 × 210 × 40 mm<sup>3</sup> is used. A releasing agent (Silicon spray) is used to facilitate easy removal of the composite from the mould after curing. The cast of each composite is cure under a load of about 50kg for 24 h before it removed from the mould. Then this cast is post cured in the air for another 24 h after removing out of the mould.

### III. MECHANICAL CHARACTERISATION

#### A. DENSITY AND VOID FRACTION

In case of hybrid composites, consisting of three components namely matrix, fibre and particulate filler, the modified form of the expression for the density of the composite can be written as

$$\rho_{ct} = \frac{1}{\left(\frac{W_f}{\rho_f}\right) + \left(\frac{W_m}{\rho_m}\right) + \left(\frac{W_p}{\rho_p}\right)} \quad (1)$$

Where, the suffix 'p' indicates the particulate filler materials. W and  $\rho$  represent the weight fraction and density respectively. The suffix f, m and ct stand for the fibre, matrix and the composite materials respectively. The actual density ( $\rho_{ce}$ ) of the composite, however, can be determined experimentally by simple water immersion technique. The volume fraction of voids ( $V_v$ ) in the composites is calculated using the following equation:

$$V_v = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}} \quad (2)$$

#### B. TENSILE STRENGTH

The tensile test is generally performed on flat specimens. During the test a uni-axial load is applied through both the ends of the specimen. The ASTM standard test method for tensile properties of fibre resin composites has the designation D 3039-76. The length of the test section should be 200 mm.

TABLE I. LIST OF FIBRE/PARTICULATE FILLED COMPOSITES FABRICATED

Designation	Composition
A	Polyester + 0 wt% RM
B	Polyester + 10 wt% RM
C	Polyester + 20 wt% RM
D	Polyester +20wt% GF+0 wt% RM
E	Polyester +30wt% GF+0 wt% RM
F	Polyester +40wt% GF+0 wt% RM
G	Polyester+40wt% GF+10wt% RM
H	Polyester+40wt% GF+20wt% RM

#### C. FLEXURAL STRENGTH

The 3-point bend test is conducted as per ASTM standard (D2344-84) using the same UTM. The data recorded during the 3-point bend test is used to evaluate the flexural strength (F.S.) using the following equation:

$$F.S = \frac{3PL}{2bt^2} \quad (3)$$

Where, P is maximum load, b the width of specimen and t the thickness of specimen and L is the span length of the sample.

#### D. MICRO-HARDNESS

Micro-hardness measurement is done using a Leitz micro-hardness tester. A diamond indenter, in the form of a right pyramid with a square base and an angle 136° between opposite faces, is forced into the material under a load of 24.54N

#### E. SCANNING ELECTRON MICROSCOPY

The surfaces of the eroded surface composite are examined directly by scanning electron microscope JEOL JSM-6480LV. The scales are washed, cleaned thoroughly, air-dried and are coated with 100 Å thick platinum in JEOL sputter ion coater and observed SEM at 20kV.

#### F. EROSION WEAR TEST

The jet erosion test rig used in this work employs one 80 mm long nozzle of 3 mm bore. This nozzle size permits a wider range of particle types to be used in the course of testing, allowing better simulations of real erosion conditions. The mass flow rate is measured by conventional method. Particles are fed from a simple hopper under gravity into the groove. Velocity of impact is measured using double disc method.

### IV. TAGUCHI EXPERIMENTAL DESIGN

Taguchi design of experiment is a powerful analysis tool for modelling and analyzing the influence of control factors on performance output. Exhaustive literature review reveal that parameters viz., impact velocity, impingement angle, fibre loading, filler content, erodent size etc. largely influence the erosion rate of polymer composites [6]. In the present work, the impact of four such parameters are studied using  $L_9(3^4)$  orthogonal design. In a conventional full factorial experiment design, it would require  $3^4 = 81$  runs to study four parameters each at three levels whereas, Taguchi's factorial experiment approach reduces it to only 9 runs offering a great advantage in terms of experimental time and cost. The parameter settings for erosion test are given in Table 2. The experimental observations are transformed into a signal-to-noise (S/N) ratio. There are several S/N ratios available depending on the type of characteristics. The S/N ratio for minimum erosion rate coming under smaller is better characteristic, which can be calculated aslogarithmic transformation of the loss function as shown below.

Smaller is the better characteristic:

$$\frac{S}{N} = -10 \log \frac{1}{n} \left( \sum y^2 \right) \text{ where } n \text{ the number of observations,}$$

and y the observed data. "Lower is better" (LB) characteristic, with the above S/N ratio transformation, is suitable for minimization of erosion rate.

## V. RESULT AND DISCUSSION

### A. DENSITY AND VOLUME FRACTION OF VOIDS

The density values of the composites calculated theoretically using (1) is not equal to the experimentally measured values. Actually, this difference is a measure of voids and pores present in the composites. The theoretical and measured densities of the composites along with the corresponding volume fraction of voids are presented in Table 3. It is found that with the increase in filler (RM) content from 0 to 20 wt%, there is an increase in density by about 11% while the void fraction or porosity increases almost twice. With the addition of red mud as the filler material, more voids are found in the composites. With the incorporation of glass fiber, the void fraction in the composites further increases.

Density of a composite depends on the relative proportion of matrix and reinforcing materials and this is one of the most important factors determining the properties of the composites. The void content is the cause for the difference between the values of true density and the theoretically calculated one. The voids significantly affect some of the mechanical properties and even the performance of composites in the workplace. Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration and weathering. It is understandable that a good composite should have fewer voids. However, presence of void is unavoidable in composite making particularly through hand-lay-up route.

### B. TENSILE AND FLEXURAL STRENGTH

The test results for tensile flexural strength are presented in Table 4. It is noticed that as the content of red mud particles increases, both the tensile as well as bending strengths of composite decline gradually. There is a drop of about 14% in the tensile strength with 20 wt% red mud filling when compared with the unfilled polyester. However, with the incorporation of glass fibre, the strength improves substantially. The drop in flexural strength with filler content is also gradual but is not as high as that in the case of tensile strength. Similar kind of observations in terms of tensile and flexural strength has been reported for different fillers [7] for fiber reinforced thermoplastics composites. It may be mentioned here that both tensile and flexural strengths are important for recommending any composite as a candidate for structural applications.

TABLE II. PARAMETER SETTING FOR EROSION TEST

Control factor	Level			Units
	I	II	III	
A: Velocity of impact	32	44	58	m/sec
B: Fibre loading (for composites D,E,F)	20	30	40	wt%
B: Filler content (for composites F, G, H)	0	10	20	wt%
C: Impingement angle	30	60	90	degree
D: Erodent size	200	300	400	μm

The corner points of the irregular-shaped red mud particles might be resulting stress concentration in the polyester matrix which is the probable reason for this decline in the strength properties of these particulate-filled composites compared with the unfilled ones. The finding that the strength decreases with increase in filler content further strengthens this phenomenon.

### C. MICRO-HARDNESS

The incorporation of red mud into the composites, the mean hardness is improved. Similar property modification has been previously reported for  $Al_2O_3$  particles reinforced in polyurethane matrix [8].

## VI. EROSION WEAR CHARACTERISTICS

### A. SURFACE MORPHOLOGY

To identify the mode of material removal, the morphologies of eroded surfaces are studied under scanning electron microscope. Fig. 1a and b present the microstructure of the composite eroded with high impact velocity (58m/sec) at an impingement angle of 60°. Fig. 1a shows local removal of resin material from the impacted surface resulting in exposure of the fibres to the erodent flux. This micrograph also reveals that due to sand particle impact on glass-fibres, there is fragmentation of the fibre body. Repetitive impacts of hard silica sand form wear debris, as seen in Fig. 1b due to brittle fracture of the fibre body as well as of the red mud particles present in the matrix body. The debris in platelet form is removed and account for the measured wear loss.

### B. STEADY STATE EROSION

Erosion wear behaviour of materials can be grouped as ductile and brittle categories although this grouping is not definitive. Thermoplastic matrix composites usually show ductile behaviour and have the peak erosion rate at around 30° impingement angle because cutting mechanism is dominant in erosion. While the thermosetting ones erode in a brittle manner with the peak erosion occurring at normal impact. However, there is a dispute about this failure classification as the erosive wear behaviour depends strongly on the experimental conditions and the composition of the target material [9]. In the present work, erosion curves are plotted from the results of erosion tests conducted for different impingement angle keeping all other parameters constant i.e. impact velocity 32m/sec and erodent size 200μm. Fig. 2 shows the dependence of the erosion rate of glass-polyester composites with different fibre content (without red mud) on the impingement angle. It can be seen that the peaks of erosion rate curves are located at an angle of 45° for all the samples irrespective of fibre content. This shows semi-ductile erosion behaviour of the composite. It is further noted that with increased fibre content the erosion rate of the composites is greater. The erosion wear rates of glass-red mud-polyester composites as a function of impingement angle ( $\alpha$ ) are shown in Fig. 3. It can be seen that filling of composite with red mud particles reduces the wear rate of the glass-epoxy composites quite significantly.

TABLE III. MEASURED AND THEORETICAL DENSITIES OF THE COMPOSITES

Composites	Measured density (gm/cc)	Theoretical density (gm/cc)	Volume fraction of voids (%)
A	1.338	1.350	0.88
B	1.403	1.429	1.81
C	1.489	1.529	2.61
D	1.451	1.493	2.89
E	1.529	1.575	2.92
F	1.604	1.672	4.06
G	1.715	1.805	4.98
H	1.827	1.957	6.64

TABLE IV. MECHANICAL PROPERTIES OF THE COMPOSITES

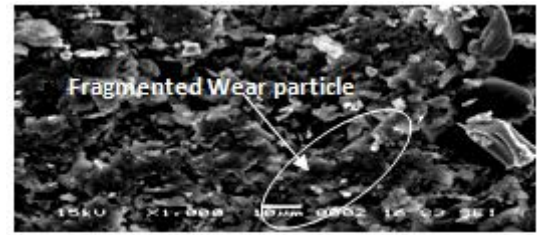
Composite Designation	Tensile Strength (MPa)	Flexural Strength (MPa)	Micro-Hardness (Hv)
A	165	135.0	25.0
B	159	165.8	28.3
C	142	158.4	31.8
D	236	217.8	32.6
E	274	253.6	34.9
F	308	296.3	36.4
G	287	259.6	42.3
H	273	234.7	47.5

The unfilled composite, shows maximum erosion occurring at  $\alpha = 45^\circ$  while for both the filled composites (with 10wt% and 20wt% RM content) the value of  $\alpha$  where the peak erosion occurs is found to be  $60^\circ$ . This shift in the erosion behaviour is an indication of loss of ductility and is obviously attributed to the presence of particulate fillers. It can be clearly seen that erosion performance of glass-epoxy composites improves with red mud filling and this improvement is a function of filler content.

## VII. EROSION WEAR ANALYSIS USING TAGUCHI EXPERIMENTAL DESIGN

The erosion wear rates conducted for all the 9 test runs along with the corresponding S/N ratio for the glass-polyester composites with and without red mud filling. The overall mean for the S/N ratio of the wear rate are found to be -48.777 dB and -47.55 dB for composites without and with red mud respectively by using the software MINITAB 14 specifically used for design of experiment applications. The S/N ratio response analyses for composites of both the types are presented in Tables 5 and 6 respectively. Similarly, the effects of individual control factor on wear rate are shown graphically in Fig. 4 and 5 for the composites D, E, F and F, G, H respectively. In both the cases, as shown from the respective S/N ratio response tables, it is clear that among all the factors, impact velocity is the most significant factor followed by filler/fibre content and impingement angle while the erodent size has the least or almost no significance on wear rate of the composites under this investigation. The analysis of the results leads to the conclusion that factor combination of  $A_1$ ,  $B_1$ ,  $D_1$  gives the minimum wear rate for the glass-polyester composites whereas factor combination of  $A_1$ ,  $B_1$ ,  $D_1$  gives the minimum erosion rate for the glass-polyester composites

with red mud filler.



(a)

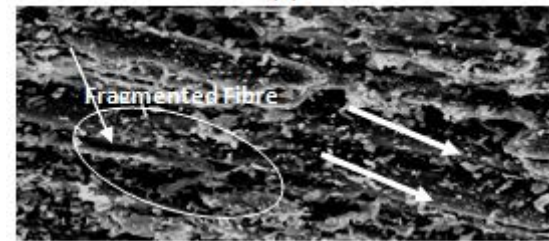
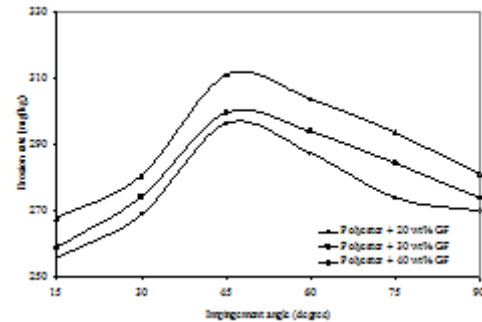

 Figure 1. SEM micrograph of glass-polyester-red mud composite eroded with erodent size 200µm at impact vel. of 58m/s and impingement angle  $60^\circ$ 


Figure 2. Erosion rate vs. angle of impingement for different fibre loading

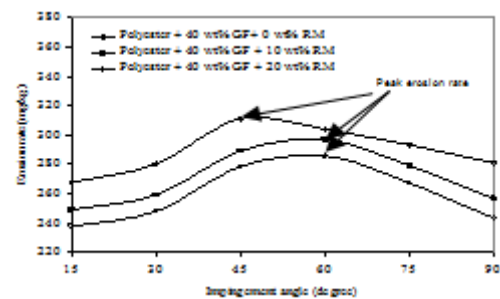


Figure 3. Erosion rate vs. angle of impingement for different red mud content

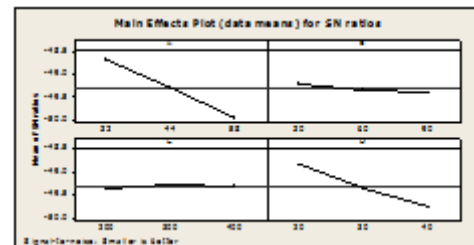


Figure 4. Effect of control factors on erosion rate of glass-polyester composites

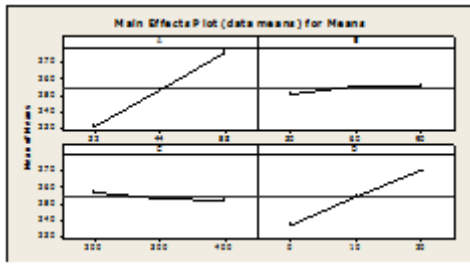


Figure 5. Effect of control factors on erosion rate of red mud filled glass-polyester composites

TABLE V. RESPONSE TABLE FOR SIGNAL TO NOISE RATIO (GLASS-POLYESTER COMPOSITES)

Level	A	B	C	D
1	-48.69	-49.22	-49.38	-48.85
2	-49.32	-49.35	-49.29	-49.36
3	-49.97	-49.41	-49.31	-49.78
Delta	1.28	0.19	0.09	0.93
Rank	1	3	4	2

TABLE VI. RESPONSE TABLE FOR SIGNAL/ NOISE RATIO (GLASS-POLYESTER-RM COMPOSITES)

Level	A	B	C	D
1	-47.28	-47.95	-48.12	-47.49
2	-48.07	-48.08	-48.03	-48.09
3	-48.82	-48.15	-48.02	-48.60
Delta	1.54	0.20	0.10	1.11
Rank	1	3	4	2

## CONCLUSION

This experimental investigation on the development, characterization and wear behaviour of red mud filled glass-polyester composites have led to the following specific conclusions:

1. Incorporation of red mud fillers decreases the tensile, flexural strengths of the glass polyester composites. However there is increase in micro-hardness and density of the composites which are also greatly depended on the content of fillers.
- 2 The presence of particulate fillers (red mud) in these composites improves their erosion wear resistance and this improvement depends on the weight content of the filler. Erosion characteristics of these composites have been successfully analyzed using Taguchi experimental design. Significant control factors affecting the erosion rate have been identified through successful implementation of this technique. Impact velocity, fibre/filler content and impingement angle in declining sequence are found to be

significant for minimizing the erosion rate of all the composites. Eroder size is identified as the least influencing control factor for erosion rate.

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## AUTHOR'S BIOGRAPHY

The author, Hemalata Jena graduated in Mechanical Engineering from Orissa School of Mining Engineering, Keonjhar in the year 2007. She has work experience in Vedanta Aluminium Ltd. By viewing problem with alumina waste she interested in work with that area. she did her postgraduate study in Mechanical Engineering with specialization in Production Engg. at the National Institute of Technology (NIT), Rourkela. Immediately after the completion of M.Tech. in 2010, she joined IIT Bhubaneswar for PhD in the department of Mechanical Engineering.

Dr Alok Kumar Satapathy is currently working as associate professor in the department of Mechanical Engineering at National Institute of Technology (NIT), Rourkela. The author has been engaged in active research in the field of composite materials and tribology. He has 150 research papers to his credit which have been published in various national and international journals of repute. He guided many M.Tech and PhD students.